

EVALUATION OF A CONTINUOUS  
ROTARY THERMAL DISTILLATION COLUMN

By

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## I. PURPOSE

Very few radical developments have taken place within the last few decades or so as far as distillation operations were concerned. Engineers have improved upon the bubble-cap column, shifted to grid trays, varied packing; however, the primary reasoning has remained the same since the advent of the Wolcott cascade. Thus, improvement after improvement has been made upon contact rectification systems. In fact, industry seems too self-satisfied with this type column, possibly because contact columns require in the majority of feasible separations low energy input and, thus, prove more economical. Rather than improvements, a radical redesign might well be in order.

Recently, there have been experimental development in regard to what is termed "thermal" distillation or rectification. This type column shows promise, particularly at low pressures. It is the purpose of this paper to present an unbiased evaluation of an experimental continuous thermal rectification column. It is the author's firm belief that such a column, as described in what follows, is both practical and feasible. With refinements, it might well compete with contact systems in certain separation problems.



## II. HISTORICAL BACKGROUND

Since the observation that components could be separated by means of a simple pot and condenser, man has undertaken the task of obtaining sharper and sharper separations. Much has been accomplished along these lines through the years. Originally the process of simple distillation was repeated step by step. With the advent of the Wolcott cascade, the requirement of individual stills and condensers became unnecessary. However, the repeated application of a reboiler and condenser is practiced in some present day molecular distillations. From the Wolcott cascade, the long step to present day columns was but a matter of refinement and convenience.

Such distillation - that is, the contacting of liquid and vapor, not in equilibrium, countercurrently - is termed contact distillation. This depends primarily upon the phenomenon that the two contacted phases attempt to approach equilibrium. The transfer of material between phases is spontaneous, and the rate of transfer depends upon the mass diffusion constants and the absolute vaporization and condensation rates at the liquid-vapor interface. Thus, we can see that an enrichment will occur.

However, another type of enrichment can be realized by means of partial vaporization and partial condensation. Either one alone or both can effect this enrichment. Let us call this thermal rectification. The interphase transfer in this case cannot be considered spontaneous, rather it is brought about by the delib-



erate addition or extraction of thermal energy. The only equilibrium consideration would be between the condensing liquid and its associated vapor or the vaporizing vapor and its parent liquid. Enrichment is brought about by the fact that the vapor formed by partial vaporization is richer and the liquid formed by partial condensation is leaner than the parent phase from which each evolved.

Several types of thermal rectifying columns have been described (1), (2), (5). One paper considers a series of heated and cooled zones associated with an unpacked tube; another employs what might be termed an "inclined" still principle; another employs directional motion into a heated zone to bring about enrichment; finally, several papers have described a rotary thermal rectification column (1), (2). Of the four types mentioned, the latter appears to be worthy of mention and experimental evaluation. Such will be the purpose of this paper. Just to mention in passing, it must be stated that in conjunction with this experimental evaluation of a rotary thermal rectifier, preliminary work is being accomplished in regard to the inclined column by the Chemical Engineering Department of Lehigh University.

A basic reflection upon all four thermal methods mentioned above should prove that they are all reversions to the old modes of multiple redistillation - the pots and condensers merely being contained in one overall unit.

Note also that, whereas, all preceding papers commented on batch type operation, this thesis will consider continuous operation.



### III. COLUMN COMPARISON

Before embarking upon a direct evaluation of the thermal rectifier, a few considerations regarding distillation in general appear to be in order.

Possibly the one variable in distillation operations which has the most adverse effect upon the system is operational pressure - the adverse effect being that brought about by pressure reduction. It is well known that the separation ability of a contact column is very poor at low pressures. In conventional stills reduction of pressure lead to the following considerations:

(1) vapor velocity increases to an extent where there is definite interference with column operation (2) the change in column pressure may be large when compared to the pressure desired at the feed point (3) the vaporization process itself becomes complicated due to hydrostatic head and surface tension phenomena. Nevertheless, these difficulties may be counteracted, invariably by improved column design.

Further adverse effects due to reduced pressure operation of contact columns are brought about by the increased diffusion rates for the vapor phase and decreased absolute condensation and vaporization rates. The former operates to both an advantage and disadvantage by means of the fact that transverse mixing is rapid and thus favorable; however, longitudinal (or vertical) mixing invariably takes place quite rapidly, and thus the vapor stream is unable to support a concentration gradient for proper column functioning.



A direct comparison between thermal and contact columns shows that low pressure enhances the operation of the former over the latter type column (1). This, to many people, is the great advantage of the thermal column over the contact.

Actually, a contact column experiences a certain degree of thermal rectification. Almost invariably a contact column is operated under adiabatic conditions, since apparently the efficiency is highest at that point. Reference (1) presents a rather fine discussion as to the fallacy behind this assumption. In fact, large deviations from adiabatic operation can lead to higher efficiencies due to the helpful effect of the thermal component.

The thermal column is considered to be independent of the majority of pressure effects which render the contact column inefficient. Operation at pressures in the vicinity of atmospheric conditions indicate that vapor stream mixing is brought about mainly by turbulence; at low pressures, by increased diffusion rates. Possibly the majority of enrichment at atmospheric conditions is brought about by partial vaporization rather than partial condensation, which is known to be a poor means of enrichment at the pressure mentioned (1).

When considering the rotary thermal rectifier one must visualize the following as taking place uniformly along the column height:

- (1) Partial vaporization of the reflux.
- (2) Blending of the above vapor with the main vapor stream.
- (3) Partial condensation of the vapor.
- (4) Blending of (3) with the main reflux stream.

These considerations are from reference (1).



#### IV. DESIGN AND CONSTRUCTION

Fundamentally the column consists of two concentric tubes, the outer one being heated and the inner one being cooled. The latter tube is also capable of being rotated so that centrifugal action can dispel the condensate formed. The basic operations of the rectifier are as stated in the latter part of section III.

In the present design the column proper is a seventy inch length of two-inch standard steel pipe, having a feed inlet at the mid-length. Surmounted around the column proper are two, four-inch flanged steam jackets, which are considered to function as rectifying and stripping reboilers. By means of proper bearing mounts and alignment, a finned type 3/4" condenser tubing is inserted between sections of 3/4" extra strong brass pipe. This serves as the column internal condenser and is rotated by a 1 H.P. electric motor.

Steam rate to the stripping section is controlled manually, while that to the rectifying section is controlled by a Fenwal thermostatic switch in conjunction with an on-off type solenoid valve. To obtain uniform temperature conditions in the above section a small amount of steam bleed is provided by cracking a by-pass valve manually. No means of steam rate measurement are provided.

Cooling water rate is measured by a mercury-water manometer and a 17/64" orifice plate. Temperature of inlet and outlet stream are measured by mercurial thermometers inserted in their respective lines.



Feed rate and distillate rate are measured by means of suitable flowrators. Bottom rate is obtained by a simple material balance. The system is termed a closed type since the bottoms and the distillate were both returned to the same tank from which the feed was pumped. Preliminary runs indicated that thermal mixing did not accomplish steady feed composition, therefore, a centrifugal pump was installed as a mixing agent.

Temperatures of the liquid reflux film were obtained from ten Cu-Constantan thermocouples, five to each section, arranged in a vertical  $180^{\circ}$  helix.

For detailed column dimension, construction, and components refer to figures 1a, 1b, and 2 in the Appendix.



## V. COLUMN THEORY

In general, the basic problem is the determination of the liquid and vapor composition,  $x$  and  $y$ , in terms of the column height  $z$  and time  $t$ . This approach can be derived easily from methods described in previous papers (1) (3). Rate equations are obtained by taking a material balance over an element of column height,  $dz$ , thus -

$$L \frac{d'x}{d'z} - hL \frac{d'x}{d't} - \phi = 0 \quad (1)$$

$$V \frac{d'y}{d'z} - hV \frac{d'y}{d't} - \phi = 0 \quad (2)$$

( $\frac{d'}{d'}$  refers to partial derivative.)

Neglecting longitudinal mixing, each equation consists of three terms: (1) the net rate of transport of the component in question into the element by flow (2) the rate of accumulation of the component in the holdup of the element (3) the net rate of transfer of the component between phases in the element. The third terms will be considered briefly in following statements.

Actually, for this paper's consideration, it is quite proper to limit the discussion to steady state operation. Let  $\phi$  be some function of the vaporization and condensation rates in the element, thus -

$$\phi = Q_v q - Q_c p$$



where  $Q_v$  and  $Q_c$  are the number of moles of vapor and condensate formed per unit length per unit time and  $q(x)$  and  $p(y)$  are the composition of the vapor and condensate formed in the column respectively. Therefore, equations (1) and (2) become -

$$L \frac{dx}{dz} + Q_{cp} - Q_v q = 0 \quad (3)$$

$$V \frac{dy}{dz} + Q_{cp} - Q_v q = 0 \quad (4)$$

Flow rates in the above equation are all considered positive, irrespective of flow directions. Considerations of flow directions for differential condensation and vaporization will be in agreement with those in reference (7) when the above equations are applied practically.

For steady state solution, subtraction of equation (3) from equation (4), followed by integration will lead to the conventional operating line equation, exactly as in contact columns (1).

$$V_y - L_x = D x_D \quad (5)$$

One must consider that in the general case,  $V$  and  $L$  in (5) above are variables and not constant. Considering that the  $Q$ 's are constant along the height of the column, as is the case in the rotary thermal still, then -

$$V = V_0 (Q_v - Q_c) z \quad (6)$$



by which the variable  $V$  can be eliminated from equations (4) and (3); therefore, proper integration leads to -

$$\frac{1}{Q_V - Q_C} \ln \left[ 1 + \frac{Q_V - Q_C}{V_0} Z_D \right] = \int_{y_0}^{y_D} \frac{dy}{Q_V Q - Q_C P - (Q_V - Q_C) y} \quad (7)$$

This equation is for the section above the feed, corresponding equations can be obtained for the liquid and for the section below the feed. A brief reflection will show equation (7) is but a modification to the Rayleigh equation (7).

For the system investigated, assumptions similar to those in reference (1) were made -

$$\begin{aligned} p(y) &= y \\ q(x) &= \frac{ax}{1 + (a-1)x} \end{aligned} \quad (8)$$

Thus, with the use of (8),  $x$  can be eliminated from (7) by means of (5). Performance can then be obtained by integration.

Considering equation (6), a simplification can be observed in cases where  $Q_C = Q_V$ , in which case  $L$  and  $V$  are constant; thus, equation (7) reduces to -

$$\frac{QZ}{V} = \int_{y_0}^{y_D} \frac{dy}{q-p} \quad (9)$$

$$\frac{QZ}{V} = \int_{y_B}^{y_0} \frac{dy}{q-p} \quad (9a)$$



Such operation is termed adiabatic (1).

By observation, the left hand members of equations (9) and (9a) are dimensionless and in this case are termed  $r$  or heat ratio.(1). Actually, they are a ratio between the column vaporization rate and vapor flow rate; or better, a measure of the number of times the material is redistilled in passing through the column. As stated in reference (1), this is a proper term to be employed in regard to a thermal column rather than transfer units, et.al..

Correlation can be had between contact and thermal columns but the differences are apparent in that one employs the basis of heat input rather than a composition consideration. Due to the nature of the equations employed for both types of columns, there is close agreement between heat units, theoretical plates, and transfer units.

Another particular case may be observed in the case of infinite heat ratio or minimum reflux. Thus, by setting the denominator of (9) and (9a) equal to zero, or -

$$Q_v - Q_{cp} = (Q_v - Q_c)y \quad (10)$$

and for adiabatic operation

$$p = q \quad (10a)$$

give formulae for minimum reflux.

As mentioned previously, the column was operated at atmospheric conditions. Thus, we must consider the assumptions and approximations which will of a necessity be valid.



Firstly, we may accept the statement that at atmospheric pressure most systems will have predominate transverse mixing due to turbulence rather than that due to diffusion in the vapor phase. This mixing is rapid and may be considered instantaneous. Then too, at atmospheric pressure, partial condensation does not contribute to enrichment appreciably and may in most cases be neglected.

Assuming constant  $a$ , the above considerations lead to -

$$p = y$$

$$q = \frac{ax}{1+(a-1)x} \quad (8)$$

From reference (1), the simplest case, adiabatic operation at total reflux, leads to -

$$r = \frac{1}{a-1} \ln \frac{y_0}{y_D} - \frac{a}{a-1} \ln \frac{1-y_D}{1-y_0} \quad (11)$$

Rearranging equation (11) -

$$r = \frac{1}{a-1} \ln \frac{y_D (1-y_0)}{y_0 (1-y_D)} + \ln \frac{1-y_0}{1-y_D} \quad (12)$$

and noting that the second right hand member is negligible relative to the first, a direct comparison between bubble plate and packed columns can be made.

For bubble plates -

$$n = \frac{1}{\ln a} \ln \frac{y_D (1-y_0)}{y_0 (1-y_D)} \quad (13)$$



and for packed columns -

$$\frac{z}{VH\phi} = \frac{1}{2} \frac{a+1}{a-1} \ln \frac{y_D(1-y_0)}{y_0(1-y_D)} \quad (14)$$

Thus, the characterization factors are -

$$\text{plate: packed: thermal} = \frac{1}{\ln a} : \frac{a+1}{2(a-1)} : \frac{1}{a-1}$$

Further cases that can be considered are partial reflux under adiabatic conditions and non-adiabatic conditions. The former leads to equations identical with those obtained by Dodge and Huffman (6). The latter leads to equations which are quite complicated algebraically and are of little immediate interest (1).

#### Definition of Terms

- $L$  = liquid flow rate  $\left[ \text{moles } T^{-1} \right]$ .  
 $V$  = vapor flow rate  $\left[ \text{moles } T^{-1} \right]$ .  
 $Q_c$  = condensation rate per unit length of column  $\left[ \text{moles } T^{-1} L^{-1} \right]$ .  
 $Q_v$  = vaporization rate per unit length of column  $\left[ \text{moles } T^{-1} L^{-1} \right]$ .  
 $p(y)$  = composition of condensate formed in column,  $\left[ \text{mole fraction light component} \right]$ . Expressed as a function of  $y$ .  
 $q(x)$  = composition of vapor formed in column,  $\left[ \text{mole fraction light component} \right]$ . Expressed as a function of  $x$ .  
 $h_L$  = liquid holdup per unit length  $\left[ \text{moles } L^{-1} \right]$   
 $h_v$  = vapor holdup per unit length  $\left[ \text{moles } L^{-1} \right]$



$$\phi = Q_v q - Q_c p$$

x = mole fraction light component in liquid

y = mole fraction light component in vapor

z = height along column [L]

D = distillate rate [moles T<sup>-1</sup>]

H<sub>φ</sub> = length characteristic of packing performance [L]

a = relative volatility

n = equivalent number of theoretical plates

r =  $\frac{Q_z D}{V}$ , heat ratio

Subscripts:

D - refers to distillate or column sectional length.

o - refers to feed.

B - refers to bottoms.



## VI. EXPERIMENTAL RESULTS

The system employed in the experimental evaluation of the designed column was that of acetone-water at atmospheric pressure. Equilibrium data were obtained from reference (8) and is graphically displayed in figure 3 and noted in Table III. Fourteen experimental determinations were conducted at feed rates of 3.28 to 0.707 moles per hour and at feed compositions ranging from 0.036 to 0.375 mole fraction acetone. Analysis of feed, distillate, and bottom streams were obtained by refractometer and density readings, with more weight accredited to the former. Surprisingly, there was accurate agreement between the two methods. Figures 4 and 5 display the relationships, refractive index versus mole fraction and density versus mole fraction.

Flow rates for the feed and distillate streams were obtained by means of flowrators inserted into the respective lines; bottom's rate was then derived by an overall material balance. Water rate for the rotary condenser was obtained by calibration of a 17/64" orifice and calibration curve plotted, see figure 6. Mass flow rates appear in Table II; and overall experimental data, in Table I.

Thermocouples were installed in the wall of the column proper, as per sectional view in figure 1b, at ten selected locations for the express purpose of obtaining liquid film temperatures; such temperatures are tabulated in Table IV. Thus, one is able to estimate the approximate stream composition within



the column at particular column heights. A typical temperature profile, showing approximate composition values, is displayed in figure 7. The temperatures employed in this plot were weighted averages covering the entire fourteen runs. This presentation is considered more suitable than individual runs since temperature fluctuation or thermocouple malfunctioning is thus minimized.

Various deductions can be made by investigating Table IV in conjunction with Tables I and III. Observation will indicate that on certain runs the upper surface of the rectifying section must, of necessity, have been void of liquid due to the high temperatures recorded. This is believed to be true of all readings in excess of 147°F. This condition is definitely true on runs 1 and 2, and possibly 7 through 12. The temperature drop on all runs at position 5 can be explained by the fact that the thermocouple was located at the bottom of the rectifying steam jacket, steam condensate was thus acting as a coolant. Due to the fact that the rectifying section was supplied steam by means of an on-off temperature controlled solenoid valve, temperature readings in this section were erratic; whereas, there is some semblance of continuity in the stripping section readings.

Evaluation of the column from the experimental data is based upon the following assumptions:

- (1) Adiabatic conditions exist.
- (2) An average latent heat of vaporization exists for both the rectifying and stripping sections.
- (3) An average relative volatility exists in both above sections.



When one considers the equations contained in Chapter V, it is apparent that such assumptions are valid. Comparison between contact and thermal columns will thus be at a minimum; the inference being that the rotary thermal rectifier is then at its lowest possible efficiency. Therefore, heat unit values will be at a minimum. However, knowing that non-adiabatic conditions probably exist, the thermal column will perform at a much better efficiency and compare more favorably with the contact column than it does at this minimum consideration.

Calculation methods for comparative values is as follows:

- (1) Solve for overall  $Q_{cz}$ ,  
 $Q_{cz} H_v \text{ ave.} = Q_w$
- (2) Set  $Q_{cz} = L$ . This consideration is brought about by an analogy between the thermal column and a contact column employing a partial condenser.
- (3) Solve for  $V$  and also  $L'$  and  $V'$ . Then employ McCabe-Thiele graphical method to obtain number of plates in rectifying and stripping sections.
- (4) Knowing  $V$  and  $V'$ , solve for  $r$  (heat ratio).
- (5) Although the use of the method outlined in steps (1) through (4) is merely an approximation, it is considered accurate enough. Otherwise the solution to equation (9) becomes quite arduous, eventually leading to the following form:



$$\frac{Q_z}{V} = \int_{y_0}^{y_D} \frac{[V-D-(a-1)DX_D] + (a-1)V_y \frac{dy}{y-(a-1)V_y^2}}{-aDX_D + [aV-V+D+(a-1)DX_D]}$$

the solution to which can be obtained in any table of integrals. However, since V would be unknown; a detailed trial and error method, involving trigonometric and logarithmic functions, must be employed.

Sample Calculation:

(1)  $F = 0.841 \text{ lb moles/hr} = 27.65 \text{ lbs/hr.}$

$D = 0.312 \text{ lb moles/hr} = 16.02 \text{ lbs/hr.}$

$B = 0.529 \text{ lb moles/hr} = 11.63 \text{ lbs/hr.}$

$X_F = 0.375 \text{ mole fraction acetone}$

$X_D = 0.840 \text{ mole fraction acetone}$

$X_B = 0.100 \text{ mole fraction acetone}$

$Q_w = 28,800 \text{ BTU/hr}$

$t_D = 141^\circ\text{F}$

$t_B = 154^\circ\text{F}$

$H_v = 16,120 \text{ BTU/lb mole, average latent heat of vaporization}$

(2)  $Q_z H_v = Q_w$

$$Q_z = \frac{Q_w}{H_v} = \frac{28,800}{16,120} = 1.785 \text{ lb moles/hr}$$

therefore,

$L = 1.785 \text{ lb moles/hr}$

$V = 2.097 \text{ lb moles/hr}$

$L' = 2.938 \text{ lb moles/hr}$

$V' = 2.097 \text{ lb moles/hr.}$



(3) Plate calculation done graphical. See figure 3.

$$n = 1.4$$

$$(4) \quad r_1 = \frac{QZ}{V} ; \quad r_2 = \frac{QZ}{V'}$$

$$r = r_1 + r_2$$

$$r = 1.705 \text{ heat units.}$$

To obtain a size comparison between a contact column and the thermal column, the following data from Perry's "Chemical Engineer's Handbook", page 621, are employed:

Fiberglass Packing

density - 4.06 lb/ft<sup>3</sup>

height - 6.5 ft

diameter - 12 in

Acetone - Water System

0.18 - 0.82 feed composition

$L = 830 \text{ lb/hr ft}^2$

HETP = 1.8

$N = 3.6$

Using the values obtained for run no. 6 from Table V, the following is derived:

(1) Making the height of the packed tower equivalent to 1.35 transfer units, thus -

$$\text{tower height} = 1.8 \times 1.35 = 2.43 \text{ ft}$$



(2) Flow rate (liquid) through thermal column:

$$\text{cross sectional area} = \frac{\pi}{4}(D_o^2 - D_i^2) = 0.955 \times 10^{-2} \text{ ft}^2$$

$$L_o = \frac{L+L'}{2} = 7.805 \text{ lbs moles/hr}$$

Assume average composition of  $L_o = 0.20-0.80$

therefore -

$$L_o = 7.805 \times 26 = 203 \text{ lbs/hr.}$$

(3) Thus -

$$L = \frac{203}{0.955} \times 10^2 = 21,200 \text{ lbs/hr ft}^2$$

(4) Therefore - a packed tower to handle that flow would have to have a diameter determined as follows:

$$\frac{L_1}{L_2} = \frac{D_1^2}{D_2^2}$$

$$D_1 = 12 \text{ in}$$

$$L_1 = 830$$

$$L_2 = 21,200$$

$$D_2^2 = \frac{144 \times 21,200}{830} = 15.9 \text{ ft}$$

Another consideration may be made in the flow rate in lbs/hr - thus,

	<u>Thermal</u>	<u>Packed</u>	<u>Modified Packed</u>
L	203	12"-830	6"-203



$$\frac{D_1^2}{D_2^2} = \frac{830}{203} = 4 \quad D_2 = 6 \text{ in}$$

Thus, the size requirements for a thermal column will be much less than a packed tower, both handling the same quantity. It must be remembered that the above comparison is but a rough approximation.



## VII. CONCLUSIONS

From an evaluation of the experimental data, the following conclusions are obtained:

- (1) The thermal column is as efficient at high feed rates and low feed compositions as is a plate column operating over the same composition conditions.
- (2) The thermal column is less efficient at low and medium feed rates and medium composition.
- (3) The thermal column is more efficient at medium feed rates and high feed composition.
- (4) Rotary condenser speed has no effect upon column operation as long as speed is such that condensate is dispelled by centrifugal action.
- (5) Changing the cooling water inlet temperature did not effectively change column operation.
- (6) Size requirements for the thermal column will be less than those of a contact column handling the same flow rate,  $\text{lbs/hr ft}^2$ .

In the above, the following definitions apply:

- (a) high feed rate - 3.0 lb moles/hr and above
- (b) medium feed rate - 1.0 to 3.0 lb/moles/hr
- (c) low feed rate - below 1.0 lb moles/hr
- (d) high feed composition - 0.35-0.40 mole fraction acetone



(e) medium feed composition - 0.16-0.19 mole  
fraction acetone

(f) low feed composition - 0.03-0.06 mole fraction  
acetone

Thus, it can be stated that the thermal column is as useful, and possibly more useful, than a plate column under a variety of system conditions. It is firmly believed that there is a definite place for the rotary thermal column in industrial practice and it is a sincere hope that further experimental projects will bring this about.



### VIII. RECOMMENDATIONS

Since this report covers but a minor investigation and evaluation of a rotary thermal column, the following recommendations are made:

- (1) Change the inlet feed connection to one which will include a distribution weir, so that feed distribution is even along the column wall. The lack of such an arrangement on the present column did not, it is believed, alter results in any appreciably amount.
- (2) Conduct an evaluation of various type materials and shapes for the rotary condenser. The present finned type allowed very little space for vapor flow - this possibly resulted in high vapor velocities and decreased the vertical concentration gradient, and therefore, the column's separating power.
- (3) Relocate thermocouples to obtain proper readings and include some means for vapor and liquid sampling. With the latter two installed, thermocouples would not be required.
- (4) Split the column mechanically so that liquid and vapor lines alone would connect the rectifying and stripping sections.
- (5) Conduct evaluation of heat transfer coefficients for the column.



- (6) Provide means of measuring steam condensate rate; thus,  $Q_c$  can be evaluated.
- (7) Employ a thermo-pressure regulator on the steam chests so that operation is continuous and steady, rather than off-on as in the present system.
- (8) Investigate the capabilities of the column in regard to close boiling systems.
- (9) Finally, evaluate the system under vacuum and also at high pressures.



TABLE I  
EXPERIMENTAL DATA

Run No.	1	2	3	4	5	6	7	8
F	3.28	3.21	3.03	3.05	2.25	2.27	0.725	0.707
D	0.05	0.06	0.085	0.12	0.294	0.285	0.236	0.234
B	3.23	3.15	2.945	2.93	1.956	1.985	0.489	0.473
NF	1.3410	1.3412	1.3454	1.3453	1.3583	1.3579	1.3627	1.3627
$\rho_F$	0.980	0.978	0.971	0.971	0.929	0.930	0.892	0.892
$X_F$	0.036	0.040	0.060	0.060	0.190	0.185	0.365	0.365
N <sub>D</sub>	1.3592	1.3598	1.3586	1.3581	1.3594	1.3596	1.3591	1.3594
$\rho_D$	0.806	0.807	0.802	0.796	0.808	0.810	0.805	0.807
$X_D$	0.805	0.800	0.840	0.880	0.790	0.78	0.810	0.800
N <sub>B</sub>	1.3390	1.3393	1.3414	1.3394	1.3511	1.3511	1.3557	1.3556
$\rho_B$	0.984	0.983	0.979	0.983	0.959	0.959	0.941	0.941
$X_B$	0.025	0.026	0.038	0.026	0.10	0.10	0.150	0.150
dp	8.0	8.0	6.0	4.0	8.4	8.4	9.0	9.0
W	1758	1758	1560	1350	1800	1800	1860	1860
T <sub>1</sub>	63.7	62.2	62.1	70.9	60.8	61.2	62.1	61.3
T <sub>2</sub>	89.2	87.8	87.8	98.6	84.2	84.2	78.8	78.8
$\Sigma W$	44,800	45,000	40,100	37,400	42,150	41,400	31,050	32,500
P	20	20	20	20	20	20	21	21
T <sub>D</sub>	132.8	132.8	132.8	132.8	132.8	134.6	132.8	132.8
RPM	1600	1600	1200	1200	1600	1600	2000	2000



TABLE I  
EXPERIMENTAL DATA

Run No.	9	10	11	12	13	14
F	0.957	0.841	1.146	1.127	1.500	1.439
D	0.265	0.312	0.325	0.326	0.320	0.319
B	0.692	0.529	0.821	0.801	1.180	1.120
NF	1.3621	1.3628	1.3621	1.3621	1.3612	1.3614
$\rho_F$	0.890	0.878	0.890	0.891	0.906	0.902
X <sub>F</sub>	0.325	0.375	0.325	0.325	0.270	0.280
N <sub>D</sub>	1.3584	1.3586	1.3578	1.3579	1.3571	1.3575
$\rho_D$	0.799	0.802	0.793	0.795	0.788	0.791
X <sub>D</sub>	0.860	0.840	0.905	0.895	0.950	0.920
N <sub>B</sub>	1.3533	1.3510	1.3506	1.3503	1.3491	1.3508
$\rho_B$	0.951	0.959	0.960	0.960	0.963	0.960
X <sub>B</sub>	0.120	0.100	0.095	0.093	0.086	0.098
dp	9.0	9.0	9.0	9.0	9.0	9.0
W	1860	1860	1860	1860	1860	1860
T <sub>1</sub>	61.2	61.5	60.3	60.1	59.5	59.7
T <sub>2</sub>	78.8	77.0	77.4	77.2	77.0	77.0
Q <sub>w</sub>	32,700	28,800	31,800	31,800	32,500	32,200
P	21	21	21	21	21	21
T <sub>D</sub>	131.0	131.0	131.0	131.0	131.0	131.0
RPM	2000	2000	2000	2000	2000	2000



TABLE I  
DEFINITION OF TERMS

$F$	=	feed rate in lb moles/hr
$D$	=	distillate rate in lb moles/hr
$B$	=	waste rate in lb moles/hr
$N$	=	index of refraction at 25.7°C and based upon Na line.
$\rho$	=	density
$X$	=	mole fraction light component
$dp$	=	pressure drop across orifice, inches of Hg.
$W$	=	cooling water rate, lb/hr
$T_1$	=	cooling water inlet temperature, °F
$T_2$	=	cooling water outlet temperature, °F
$Q_w$	=	heat transfer rate, BTU/hr
$P$	=	steam chest pressure, lbs/in <sup>2</sup>
$T_D$	=	distillate temperature of Fenwal setting, °F
RPM	=	rotational speed of internal condensing tube



TABLE II  
MASS FLOW RATES, LBS/HR

Run No.	F	D	B
1	63.80	2.50	61.30
2	63.00	3.00	60.00
3	61.70	4.30	57.40
4	62.20	6.50	55.70
5	57.40	14.40	43.00
6	57.50	13.85	43.65
7	23.60	11.85	11.75
8	23.00	11.65	11.35
9	29.65	13.89	15.76
10	27.65	16.02	11.63
11	35.50	17.60	17.90
12	34.90	17.52	17.38
13	43.15	20.90	25.25
14	41.90	17.36	24.54

F = feed rate, lbs/hr

D = distillate rate, lbs/hr

B = waste rate, lbs/hr



TABLE III  
EQUILIBRIUM DATA, ACETONE-H<sub>2</sub>O SYSTEM  
PRESSURE, 760 MM. HG.

Mole Fraction C <sub>3</sub> H <sub>6</sub> O		Temp. °F
x	y	
0.000	0.000	212.0
0.015	0.325	193.3
0.036	0.564	174.9
0.074	0.734	154.9
0.175	0.800	146.7
0.259	0.831	142.0
0.377	0.840	140.9
0.505	0.849	139.8
0.671	0.868	138.2
0.804	0.902	136.6
0.899	0.938	135.3
0.930	0.960	134.1

x = mole fraction C<sub>3</sub>H<sub>6</sub>O in liquid phase

y = mole fraction C<sub>3</sub>H<sub>6</sub>O in vapor phase



TABLE IV  
LIQUID FILM TEMPERATURES, °F

Run No.	Thermocouple Position									
	1	2	3	4	5	6	7	8	9	10
1	178	180	194	183	151.5	172.5	out	182.5	194.5	200
2	175	180	187.5	182.5	150.5	167.5	"	178.5	194.5	200.5
3	131.5	129.5	129.5	134.5	129.5	157	"	169	189	198.5
4	131.5	129.5	129.5	133.5	130.5	159	"	183	197	201
5	128.5	131.0	131.5	133	134	147	"	155.5	165.5	183
6	133.5	130.5	131	135.5	133	148	"	153	165	176
7	154.5	157	159	160.5	145	158	"	167	197	203.5
8	154	157.5	159	161	147	159.5	"	172	198.5	203.5
9	155	153	145.5	150	137	148	"	166	199	203
10	154	150.5	149.5	151.5	139	154	"	170.5	200.5	202.5
11	152	146	145.5	150	138	148	"	162	198	204
12	152	147.5	146	151	138	147.5	"	163	199	203.5
13	148	142	147	152	142	149	"	160	187	201.5
14	144	142	148	151.5	141	150	"	161	189	201.5
Ave.	144.3	148.3	150.2	152.1	140.0	154.6	-	167.4	190.4	198.7



TABLE V  
COMPARATIVE VALUES OF  $n$  AND  $r$

Run No.	$t_D$	$t_B$	$H_V$	$Q_w$	$V$	$L$	$L'$	$n$	$r$
1	146.5	184.0	15,750	44,800	2.892	2.842	6.172	1.93	1.966
2	146.7	184.0	15,775	45,000	2.912	2.852	6.122	1.94	1.958
3	141.0	174.5	15,715	40,100	2.632	2.547	5.662	1.96	1.936
4	137.0	184.0	15,615	37,400	2.528	2.408	5.578	1.99	1.906
5	147.0	154.0	15,750	42,150	2.970	2.676	5.220	1.36	1.802
6	146.7	154.0	15,785	41,400	2.910	2.625	5.180	1.35	1.804
7	145.0	151.0	15,575	31,050	2.229	1.993	2.954	1.23	1.788
8	146.7	151.0	15,605	32,500	2.315	2.081	3.022	1.23	1.798
9	139.0	152.5	15,550	32,700	2.367	2.102	3.324	1.88	1.776
10	141	154	16,120	28,800	2.097	1.785	2.938	1.82	1.702
11	136.4	154	15,515	31,800	2.373	2.048	3.519	2.61	1.726
12	136.8	154.5	15,540	31,800	2.371	2.045	3.498	2.49	1.726
13	134.5	155.5	15,425	32,500	2.426	2.106	3.926	3.93	1.736
14	135.5	154.0	15,460	32,200	2.402	2.083	3.841	2.91	1.734

(1) temperature in  $^{\circ}\text{F}$

(2)  $H_V$ , average latent heat of vaporization, BTU/lb mole

(3)  $Q_w$  in BTU/hr

(4)  $V$  in lb moles/hr; also,  $V = V'$

(5)  $L$  and  $L'$  in lb moles/hr

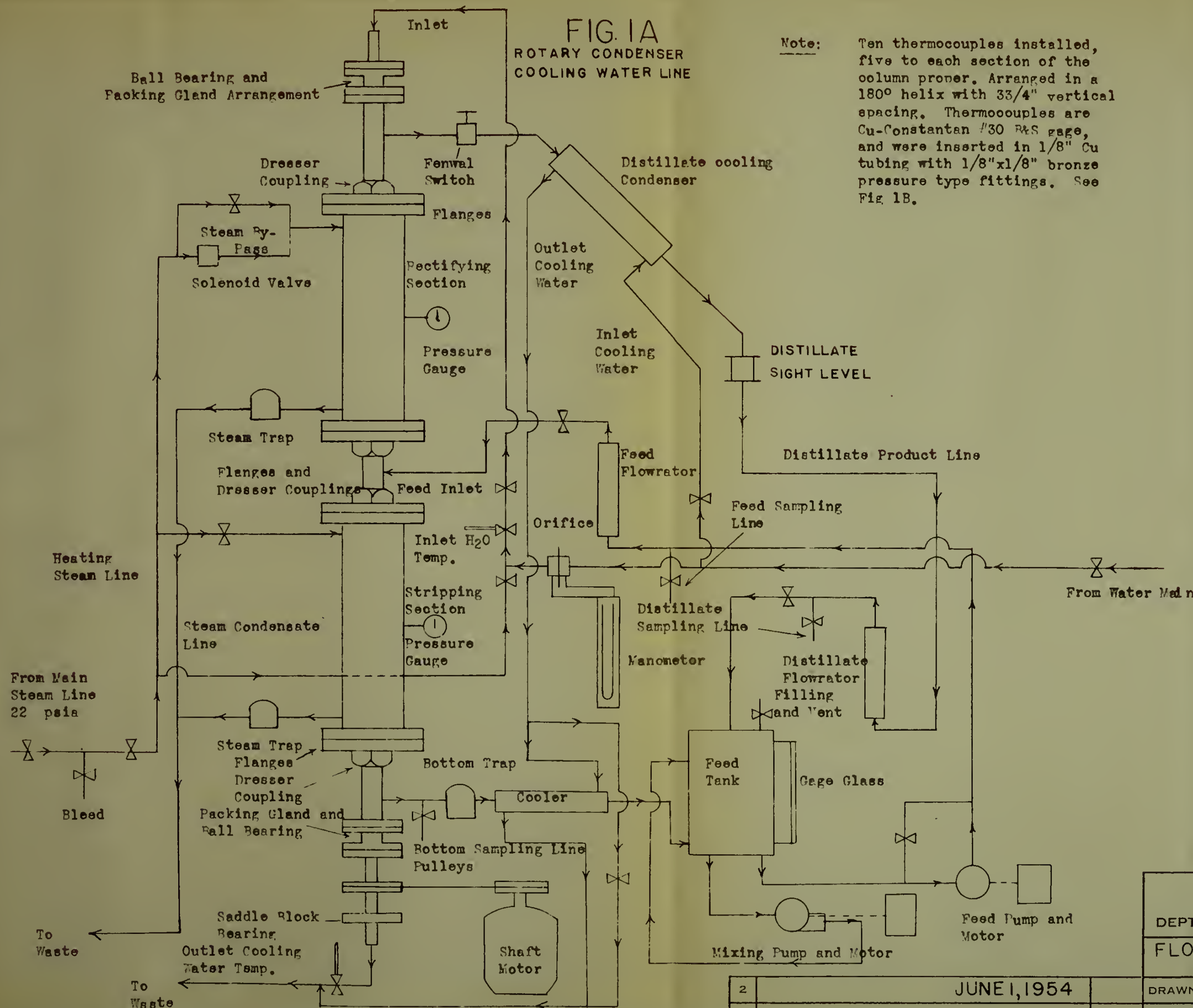
(6)  $n$ , number theoretical plates

(7)  $r$ , number heat units



FIG. 1A  
ROTARY CONDENSER  
COOLING WATER LINE

Note: Ten thermocouples installed, five to each section of the column proper. Arranged in a 180° helix with 33/4" vertical spacing. Thermocouples are Cu-Constantan #30 R&S gage, and were inserted in 1/8" Cu tubing with 1/8"x1/8" bronze pressure type fittings. See Fig 1B.



Component	Material	Size	No.
Column Proper	Steel	70"x2"	1
Rotary Condenser:			
Finned Tubing	Cu	18"x <sup>3</sup> / <sub>8</sub> "	2
Support, center	Brass	12"x <sup>3</sup> / <sub>8</sub> "	1
Support, end	Brass	20"x <sup>3</sup> / <sub>8</sub> "	2
Steam Jackets	Iron	18"x4"	2
Flanges:			
Jacket	Cast iron	9 <sup>1</sup> / <sub>2</sub> "x2"	8
Packing Gland	Cast iron	4 <sup>1</sup> / <sub>2</sub> "x2"	4
Ball Bearings:			
End		4 <sup>1</sup> / <sub>2</sub> "x1"	2
Saddle Block		4 <sup>1</sup> / <sub>2</sub> "x1"	1
Couplings, Dresser	Cast iron	2"	4
Steam Traps			3
Solenoid			1
Fenwal Thermal Regulator			1
Flowrators	max.	0.148 gpm	2
Feed Tank	Stainless	5 gal.	1
Sight Level			1
Sight Glass			1
Pressure Gauges		0-100 psi	2
Condensers, all shell and tube			
Distillate	Steel	26"x2 <sup>1</sup> / <sub>2</sub> "	1
Bottoms	Steel	34"x2"	1
Orifice	Al	17/64"	1
Manometer	Hg-H <sub>2</sub> O	36"	1
Motors:			
Feed Pump		1/3 HP	1
Mixing Pump		1/2 HP	1
Rotary Condenser		1 HP	1
Pumps:			
Feed, geared			1
Mixing, centrifugal			1
Thermometers		-10 to 110°C	2
Valves:			
Brass		3/4"	4
Brass		1/2"	6
Brass		1/4"	6
Stainless Steel		1/4"	2

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FLOW DIAGRAM, ROTARY THERMAL COLUMN

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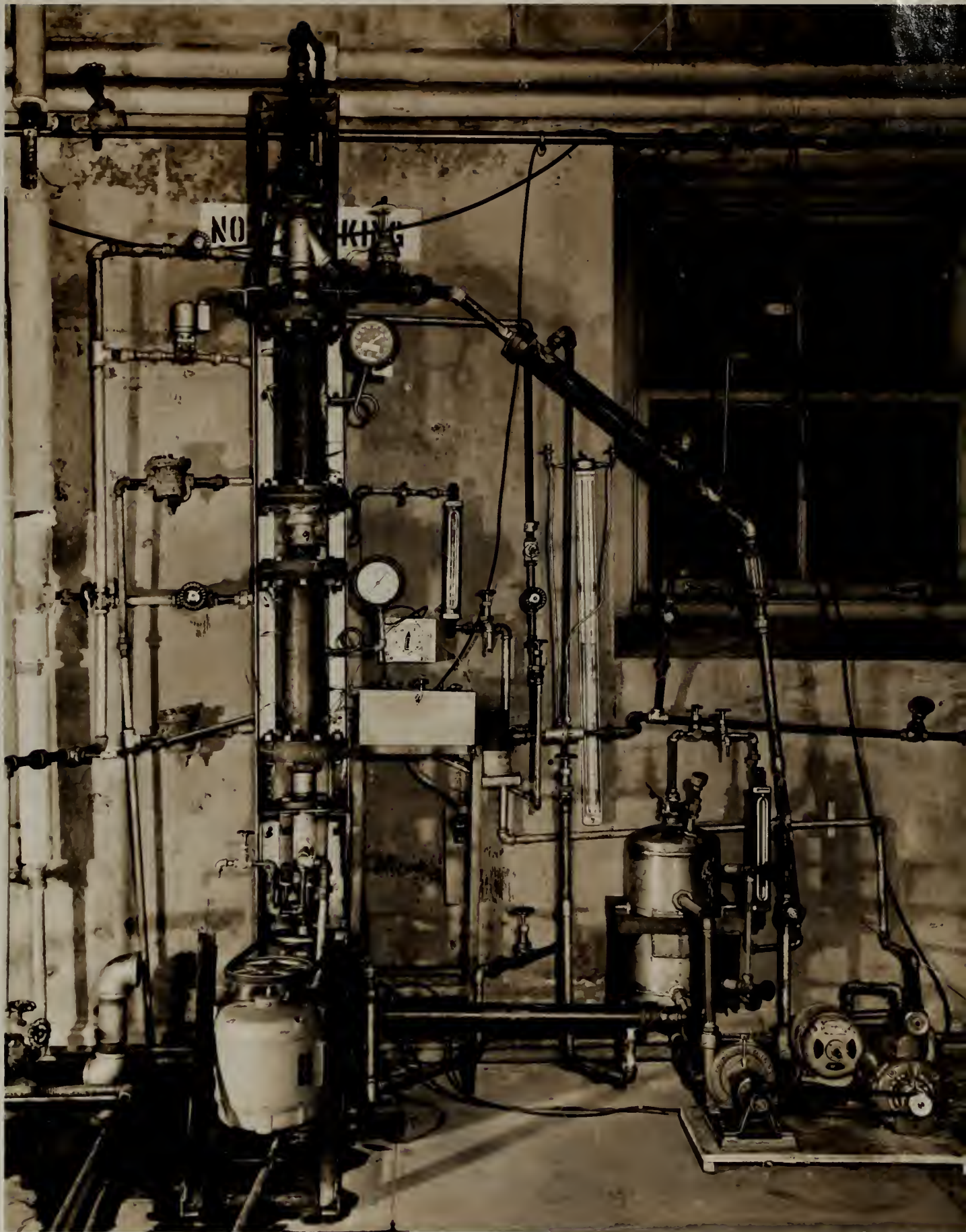
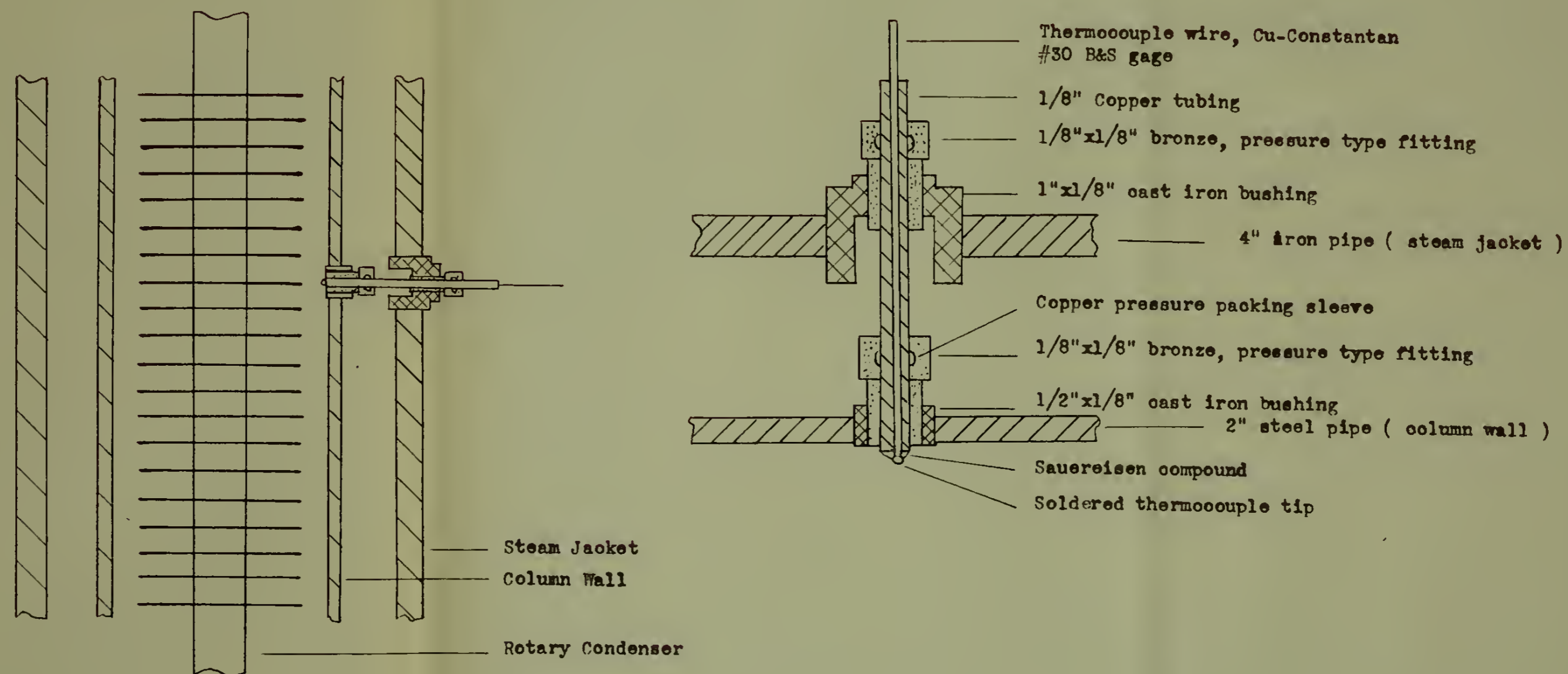


FIG. 2



FIG. 1B



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SCHEMATIC OF THERMOCOUPLE INSTALLATION

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FIG. 3

ACETONE - WATER SYSTEM  
EQUILIBRIUM DIAGRAM  
DATA FROM REFERENCE (8).

MOLE FRACTION ACETONE IN VAPOR, Y

1.0  
0.8  
0.6  
0.4  
0.2  
0.0

0.0 0.2 0.4 0.6 0.8 1.0

MOLE FRACTION ACETONE IN LIQUID, X

CHEM. ENG. DEPARTMENT  
LEHIGH UNIVERSITY  
BETHLEHEM, PA.

EQUILIBRIUM DIAGRAM

BY DATE: MAY 10, 1954  
R. L. BRADY, JR.

0.0



FIG. 4  
REFRACTIVE INDEX VS MOLE FRACTION  
ACETONE - H<sub>2</sub>O SYSTEM

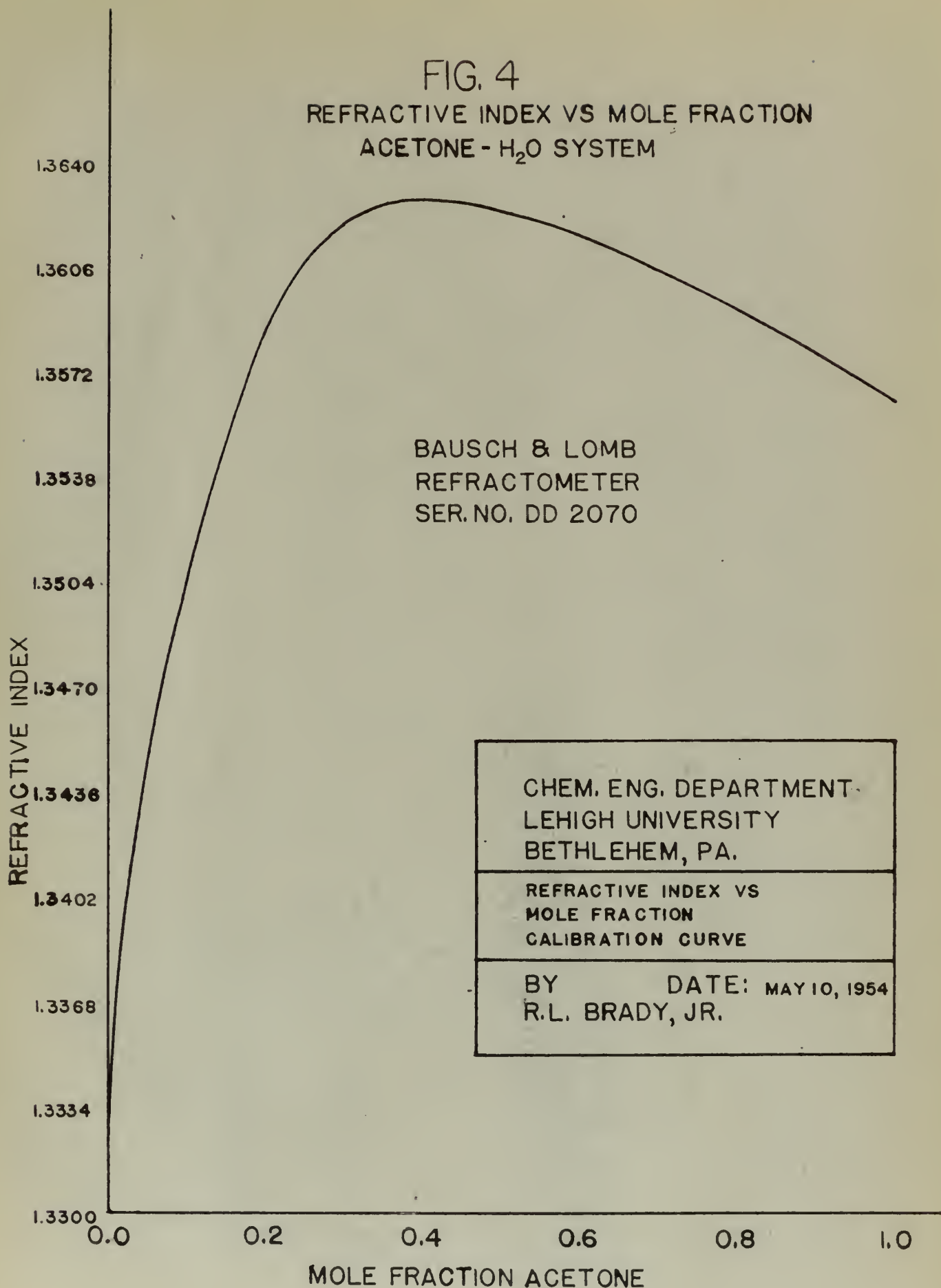




FIG. 5  
ACETONE-H<sub>2</sub>O SYSTEM  
DENSITY VS MOLE FRACTION

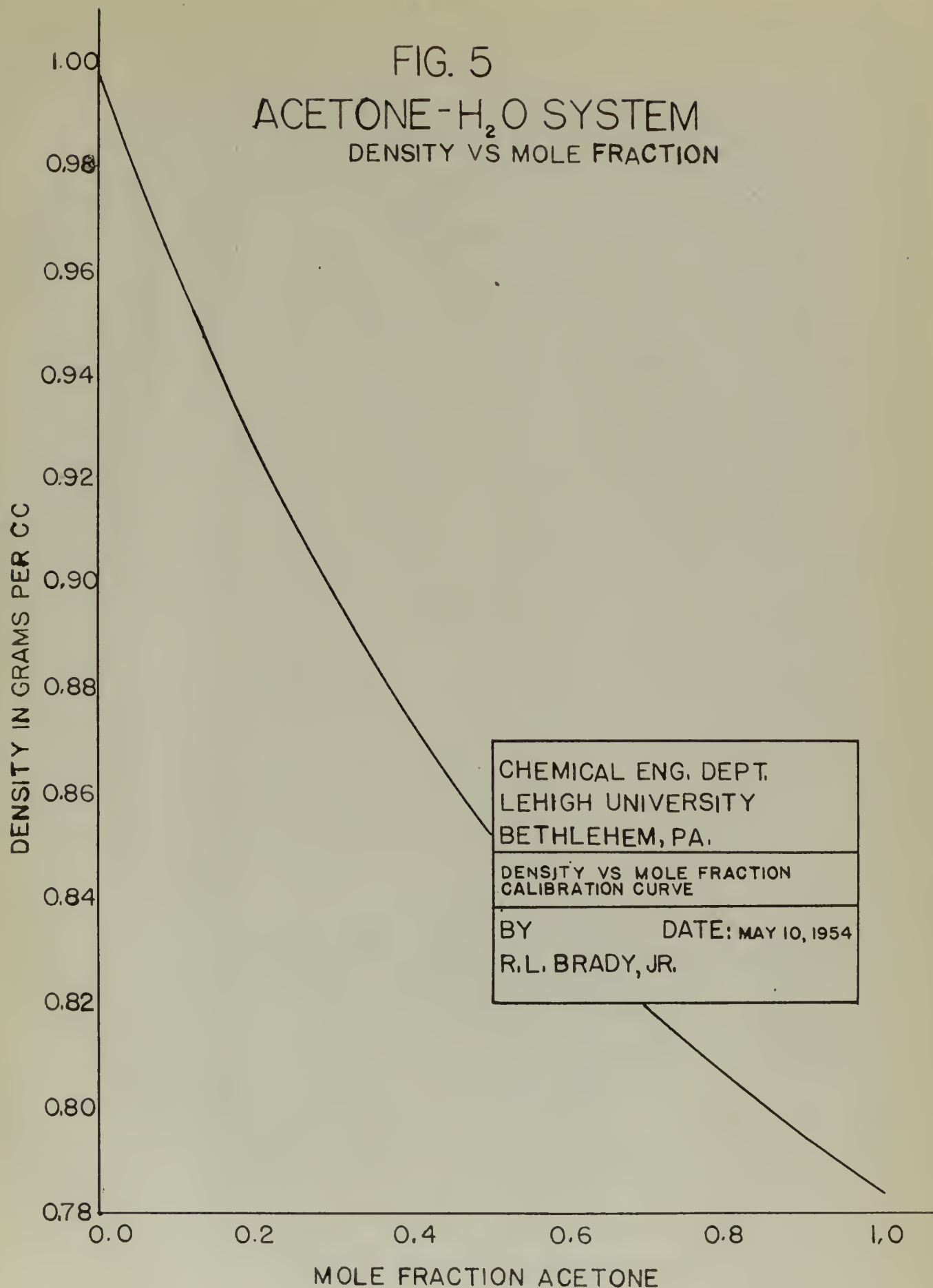




FIG. 6  
WATER FLOW RATE RELATION  
PRESSURE DROP VS WATER FLOW  
RATE

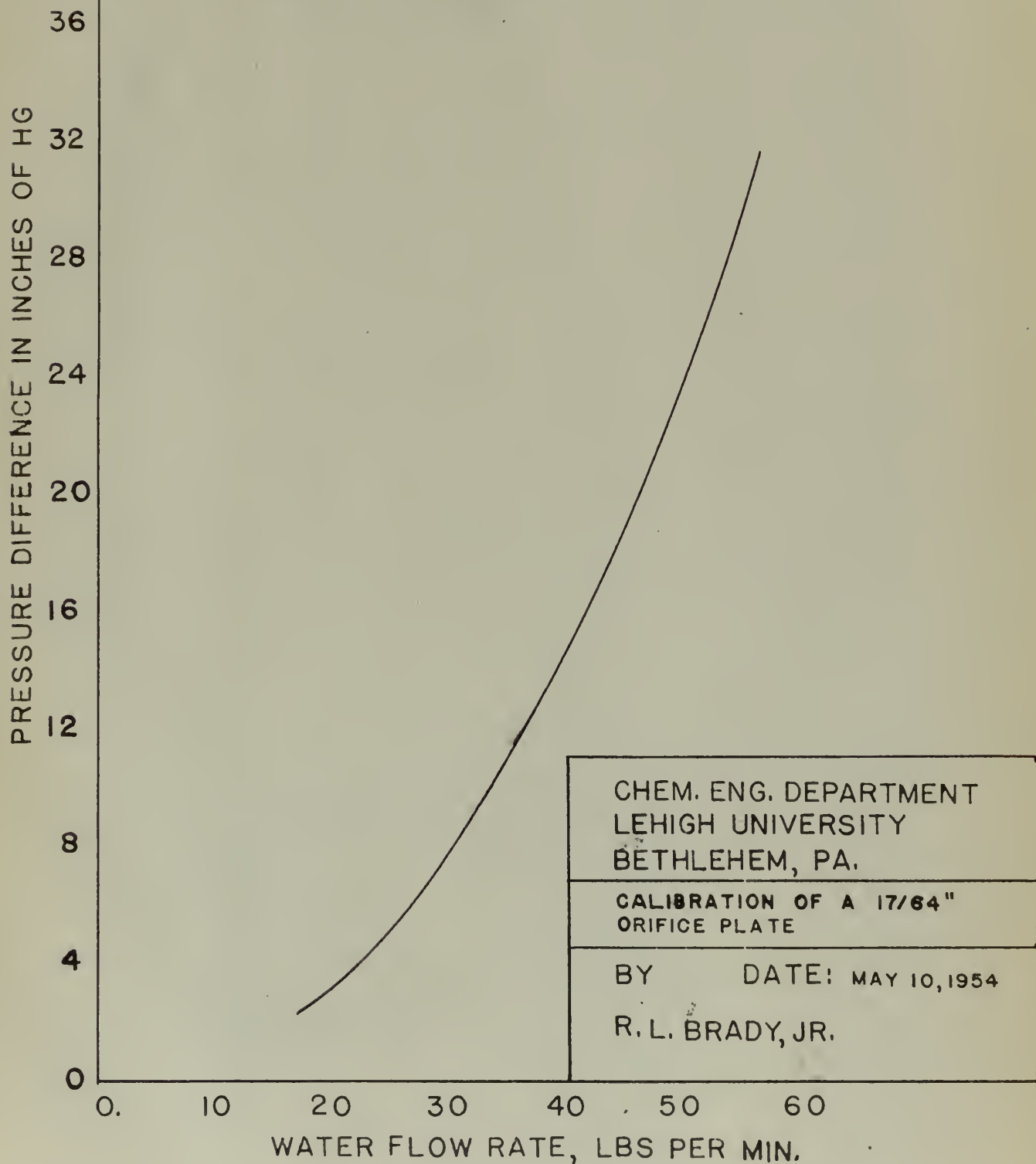
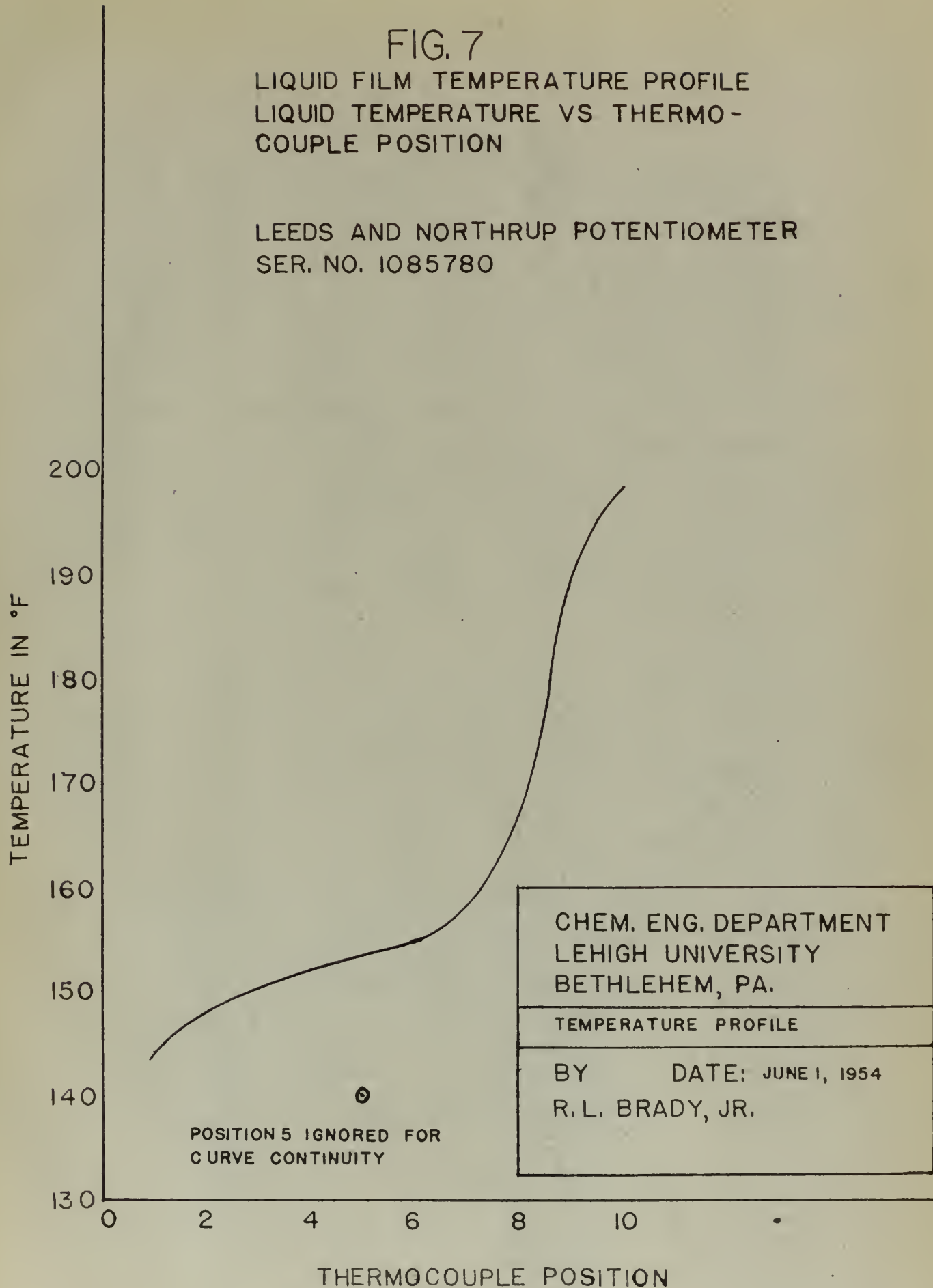




FIG. 7

LIQUID FILM TEMPERATURE PROFILE  
LIQUID TEMPERATURE VS THERMO-  
COUPLE POSITION

LEEDS AND NORTHRUP POTENTIOMETER  
SER. NO. 1085780





AUTHOR'S "VITA"

Robert Louis Brady, Jr., the son of Mr. and Mrs. Robert L. Brady, Sr. was born April 2, 1922 in Brighton, Mass. The majority of his early life was spent in the Philadelphia area, having attended the school system of Upper Darby Township. In 1940 he attended Ursinus College, planning to major in Chemistry. Due to being a member of the U.S. Naval Reserves, Brady was called to active duty with the naval service. In 1942 he passed the competitive examinations for the U.S. Naval Academy from which he graduated in June 1945, having received the degree of Bachelor of Science and being commissioned an ensign in the U.S. Navy.

After tours of duties in surface craft and submarines, the author requested graduate study assignment. In 1951 he entered the U.S. Naval Postgraduate School for a course of instruction in Chemical Engineering. After two years study, receiving the degree of Bachelor of Science, Brady registered for the graduate School, Lehigh University, pursuing a course of study leading to a Master of Science degree in Chemical Engineering.

The author is married to the former Ruth McNeal Herring of Towson, Md. and they are the parents of two sons and one daughter. After graduation Lt. Brady will be assigned to the Key West, Fla. area as engineering officer of a submarine squadron.



BIBLIOGRAPHY

- (1) A Rotary Thermal Rectifying Column - Edgar S. Byron, John R. Bowman, and James Coull. Ind. Eng. Chem. vol. 43, No.4, pp. 1002-1010.
- (2) Rotary Condenser Fractionating Vacuum Still - F.C. Benner, A. Dinardo, and D.J. Fobin. Ind. Eng. Chem. vol. 43, No. 3, pp. 722-727.
- (3) Theory of the Performance of Packed Rectifying Columns - John R. Bowman and R.C. Briant. Ind. Eng. Chem. vol. 39, No. 6, pp. 745-751.
- (4) Simplified Calculation of Diffusional Processes - Allan P. Colburn. Ind. Eng. Chem. vol. 33, No. 4, pp. 459-467.
- (5) A High-Vacuum Rectifying Column Employing Multiple Redistillation - Robert M. Schaffner, John R. Bowman, and James Coull. Trans. Am. Inst. Chem. Engrs. vol. 39, pp. 77-91 (1943)
- (6) Calculation of the number of Theoretical Plates for a Rectifying Column - Barnett F. Dodge and John R. Huffman. Ind. Eng. Chem. vol. 29, No. 12, pp. 1434-1436
- (7) Unit Operations - George Granger Brown and Associates, Wiley (1950) Selected sections.
- (8) Acetone - H<sub>2</sub>O System, Equilibrium Data - D.F. Othmer and R.F. Benenati. Ind. Eng. Chem. vol. 37, pg. 299 (1945).
- (9) Chemical Engineers' Handbook - John H. Perry editor, McGraw-Hill, third edition, 1950.







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